IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

HASSAN Y. ELNAGAR, ET AL.	
APPN. NO.: 09/484,844	**GROUP ART UNIT: 1626**
FILED: JANUARY 18, 2000	
PROCESS FOR PRODUCING N-HALOGENATED ORGANIC COMPOUNDS	**EXAMINER: LAURA STOCKTON**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 CFR 1.312

Jeffrey Todd Aplin hereby declares as follows:

I am a chemist employed by Albemarle Corporation, the Assignee of the above entitled application by unrecorded Assignment. In 1991 I received a B.S. degree from University of Mobile and in 1997 I received a PhD degree in chemistry from University of Texas at Austin. I have been continuously employed by Albemarle Corporation in a research and development capacity since December 1997.

I conducted the synthesis work reported herein at the research laboratories of Albemarle Corporation in Baton Rouge, Louisiana. The particle size determinations were conducted by co-employees in the analytical group of Albemarle Corporation at Baton Rouge, Louisiana.

The work I was requested to perform was to repeat as closely as reasonably possible Example III in Column 4 of Cole U.S. Pat. No. 4,621,096 and Example 10 in Column 5 of Waugh et al. U.S. Pat. No. 3,121,715 and to obtain a determination of the

average particle size and particle size distribution of the products directly formed in these respective examples.

Reproduction of Example III of U.S. Pat. No. 4,621,096

Since this example as given in the patent was conducted on a large scale in a 2000 gallon reactor, the reaction was scaled down to a laboratory scale operation in which all proportions specified in the patent were maintained but on a smaller scale. In particular, water (225 mL) and dimethylhydantoin (51 g) were added to a 2 L round bottom flask. Bromine (63.7 g) and an aqueous slurry of 14.75 g $Ca(OH)_2$ were added at a rate to maintain the reaction temperature below 25°C. The pH was monitored using a freshly calibrated meter and maintained between 6.8 and 7.0. After the bromine addition, 28.2 g of chlorine were bubbled into the mixture along with an aqueous slurry of 14.75 g of $Ca(OH)_2$ while maintaining the temperature below 25°C and the pH between 6.8 and 7.0. The slurry was filtered and ½ of the wet cake was set aside and labeled 8875-93A. The remaining ½ of the product was reslurried in three, 1000 mL portions of cold water and refiltered. The washed product was labeled 8875-93B. The solids were allowed to air dry for a short time then placed in an oven at 60°C overnight. The isolated weight of 8875-93A was 51.9 g, and the isolated weight of 8875-93B was 55.9 g (94.6% yield combined).

Reproduction of Example 10 of U.S. Pat. No. 3,121,715

This example was reproduced on the same scale as reported in the patent. In particular, sodium carbonate (45 g) was dissolved in 200 mL of water and cooled to 20°C with ice. Dimethylhydantoin (51 g) was then added followed by sufficient ice to give a temperature of about –5°C. Bromine (21 mL) was then added over 22 minutes while the reaction temperature was maintained below 10°C with ice. Chlorine (28.5 g) was bubbled into the mixture over 45 minutes and the foamy mixture was again maintained below

10°C with ice. The mixture was stirred for 5 minutes following the chlorine addition then filtered and washed with ice water. The product was left standing to air dry then oven dried at 60°C. A total of 95.9 g (84% yield) of product were isolated. The sample from this experiment was labeled 8875-92.

All three samples from the above experiments were submitted to the analytical group for particle size analysis. The following tabulations summarize the results that were reported to me by the analytical group relative to these samples.

Sample 8875-93A in accordance with U.S. Pat. No. 4,625,096

Particle Size Category	Result
Average Particle Size	35.69 microns
90% are this size or smaller	59.14 microns
75% are this size or smaller	44.89 microns
50% are this size or smaller	32.51 microns
25% are this size or smaller	22.09 microns
10% are this size or smaller	12.88 microns
Particle Size Range	0.040 to 282.2 microns

Sample 8875-93B in accordance with U.S. Pat. No. 4,625,096 (washed product)

Particle Size Category	Result
Average Particle Size	27.33 microns
90% are this size or smaller	45.13 microns
75% are this size or smaller	34.64 microns
50% are this size or smaller	24.62 microns
25% are this size or smaller	15.70 microns
10% are this size or smaller	7.669 microns
Particle Size Range	0.040 to 541.9 microns

Sample 8875-92 in accordance with U.S. Pat. No. 3,121,715

Particle Size Category	Result
Average Particle Size	18.48 microns
90% are this size or smaller	36.94 microns
75% are this size or smaller	20.00 microns
50% are this size or smaller	12.48 microns
25% are this size or smaller	6.914 microns
10% are this size or smaller	1.859 microns
Particle Size Range	0.040 to 282.1 microns

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the above-identified patent.

Jeffrey Todd Aplin

6/20/2003
Date